Report of the Working Group II on Chamber Coating and Treatments

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The Working Group II on Chamber Coating and Treatment met for Thursday afternoon, Dec. 11th and briefly on Friday morning. Participants are listed in the Appendix. During this group's meetings, several topics were discussed including:

- Titanium nitride coating and the achievable secondary electron yield (SEY)
- Non-evaporable getter coating process and the expected improvement in SEY and electron stimulated desorption (ESD)
- Other type coatings
- Chamber treatment such as cleaning, vacuum firing and in-situ bake

A summary of the discussions follows.

Titanium Nitride Coating

Titanium nitride (TiN) coating has been applied to radiofrequency cavities and windows to minimize the resonant electron multi-pacting effect. It was also applied to PEPII positron ring aluminum vacuum chambers to reduce the beam-induced multi-pacting and the resulted electron cloud effect. The measured secondary electron yields (SEY) of TiN coated surface vary with the coating parameters, the pretreatment and the beam conditioning (scrubbing). Only the peak SEY value, which occurs around primary electron energy of 300-500 eV, is quoted hereon. There are a few facilities available to measure SEY, with primary electron energy ranged from 50 eV to a few keV. The commonly accepted SEY for TiN coated surface is around 1.6 for as-received surface, i.e. without in-situ baking or electron scrubbing, which is significantly lower than SEY of ~ 2 for stainless and copper, and of ~ 3.5 for aluminum.

Recent coating development at BNL for Spallation Neutron Source ring vacuum chambers has found that TiN coatings done at different sputtering pressures have different SEY. Sputtering coating at lower pressure of ~ 1.5 mTorr (~ 90% argon and ~10% nitrogen) produces a bright gold color with SEY ranged from 2 to 2.5, while coating at higher pressure of ~ 5 mTorr tends to have a darker color with SEY ranging from 1.5 to 1.9. Scanning electron microscope imaging shows that the darker ones have a rough surface while the brighter ones have a smooth surface. Significant different SEY values have also been reported for other types of material with different surface roughness. Rougher surface will have lower SEY values, for example, polished copper has SEY of 2.4 while heavily oxidized copper has SEY of 1.1. The drawback of the rough surface is the initial thermal outgassing could be much higher than that of a smooth one. The presence of impurity during coating will also affect the achievable SEY, since titanium oxides and carbides may form on the surface instead of TiN.

It was agreed among the participants that a systematic measurement of SEY by various facilities, of samples prepared using different coating parameters will help clearing up the discrepancy in literature. Currently, there are SEY measurement facilities available at CERN, KEK and SLAC. The effect of beam scrubbing on SEY values was briefly discussed. Significant reduction in SEY is achieved at CERN, KEK and SLAC after electron dosage of ~ mC/mm^{2}. However, no conclusion was reached, since the SEY for technical surfaces in particular machines can vary greatly depending upon the history of beam scrubbing, venting, etc.

Non-Evaporable Getter Coating of Vacuum Chambers

This working group spent a great deal of time discussing the non-evaporable getter (NEG) coating of the vacuum chamber wall. NEG coating of an alloy of titanium, zirconium and vanadium (Ti/Zr/V of approximately equal proportion) with lower activation temperature was recently developed by Benvenuti's group at CERN. NEG coating can reduce the surface outgassing and to provide distributed pumping for the conductance limited accelerator vacuum chambers. NEG coated undulator chambers have been successfully operated in a few electron storage rings, i.e. ESRF (Grenoble, France), ELETTRA (Trieste, Italy) and SRRC (Hsinchu, Taiwan). Due to the LHC chamber coating schedule, Chiggiato of CERN couldn't attend to give the invited talk on NEG coating. Two participants, Li of Cornell and Kerseven of ESRF shared with us their experience in the development and testing of the NEG coating.

This working group also spent time reviewing the NEG coating process. Most coating were done with both chamber and the Ti/Zr/V cathode hanging vertically. The total vertical space needed for efficient production coating is approximately twice of the chamber length, i.e. 10m ceiling height for a 5m long chamber. Three pure wires of Ti, Zr and V are twisted together to form the cathode and powered at a few hundred volts DC. The coating is carried out in krypton (Kr) gas at a pressure of ~2×10^{-2} Torr, using DC magnetron sputtering with external solenoid to provide the axial magnetic field of >500 gauss. Argon gas was tested at Cornell as coating media but abandoned due to evidence of argon trapping and subsequent release during baking and activation. Coating can be done with chamber surface at 100C or 300C, with later producing a much rougher surface. The rougher surface will provide more pumping capacity before saturation therefore longer period between activation, with the drawback of few activation cycles available before reaching its lifetime capacity. The rougher surface may also have lower SEY than that of a smoother one. The achievable coating thickness is 1-2 μm as limited by the sputtering rate and the cathode heating. For example, it takes about ten hours at each solenoid position to obtain this thickness, and will require over 50 hours of continuous sputtering to coat a 5m chamber with a 1m solenoid. There may be issues with adhesion once the coating becomes too thick. Cornell has also observed film aging and peel-off after NEG was loaded with excessive hydrogen sorption.

Test at CERN indicated the coating surface is rougher if the chamber surface is rougher, such that coating on copper and stainless steel has a smooth surface, whereas coating on aluminum and beryllium has a granular structure. The pumping capacity for CO of the rough surface was found to be two orders of magnitude higher than that of the smooth surface. Tests performed at Cornell and ESRF shows that impurity trapping during coating will be detrimental to the pumping speed and capacity of NEG. NEG coating will not pump with oxygen impurity of a few percents. The activation temperature of this low temperature NEG coating was also of great interest since the achievable activation temperature will be limited by the chamber material. Pumping was observed after 150C activation at Cornell, while the aluminum chamber was activated around 180C at ESRF without any adverse effect. RHIC uses 250C x 2hr as activation, while LHC plans to use 200-300C range for activation. ESRF has used ion pumps to remove hydrogen during re-activation. Impedance of the NEG coating was also discussed and found to be ~ 50 μΩ-cm as measured at ESRF, better than stainless steel but not as good as aluminum. This impedance should be ok for most synchrotron radiation sources and hadron storage rings but may not be sufficiently low for B factory chambers.

The potential of NEG coating to reduce SEY and the electron stimulated desorption (ESD) is a relatively new development attracting considerable attention as a possible cure for electron cloud and pressure rise. Measurement at CERN indicates that NEG coating may reduce ESD from 10^{-2} for an un-coated surface to 10^{-4} for a coated one. No solid evidence is available for the reduction of ion induced desorption (ID), but the activated NEG should provide high linear pumping speed which will alleviate both ESD and ID phenomena, if not cure it. Needless to say, accelerator personnel are very interested in any observations and operating experience with NEG coating, especially the SEY values. The newly installed NEG coated beam pipes in RHIC may provide some data during the current physics run. Measurement at CERN indicates that the as-received SEY values of NEG coating range from 1.7 to 2.0, and drop to 1.1 to 1.3 after activation, and remain below 1.4 even after pumping saturation. However, SLAC reported that SEY of NEG sample in an UHV chamber went from ~1.3 to ~1.7 in 22 days, at variance with the experience at CERN. Again as for TiN coating, systematic SEY measurement of NEG coated surface, rough versus smooth, on different material, on different saturation/activation cycles and due to electron scrubbing, is needed.

The newly published data of Benvenuti on a palladium (Pd) overlayer on NEG coated surface was also discussed. This Pd layer may prevent oxide diffusion into NEG bulk during activation therefore preserve the NEG capacity for hydrogen. The pumping of hydrogen and CO by Pd is reversible by activation at low temperature of ~100C. It is very easy to sputter Pd, but the electrode is very expensive. The participants are looking forward to the development of this Pd coating into a mature technology. Diamond like graphite coating was also briefly discussed due to its low SEY, but it may be difficult to implement the coating in a long narrow chamber.

The following is the expected NEG coating development work to be down at a few labs.

Cornell – verify NEG coating's capacity for hydrogen with 10^{-13} Torr equilibrium pressure.
ESRF – will test the Pd overlayer coating in the near future.
GSI – plan to coat SIS dipole chambers with NEG.
KEKB – may test NEG without activation for SEY on a straight section in positron ring.
LHC LSS & LEIR – will bake chambers at 150C then activate the NEG coating at 200-300C.
PEPII – will TiN coat the LER straight section stainless steel chambers to reduce SEY.
RHIC – will test the newly installed NEG coated pipes under various beam conditions.

Chamber Cleaning and Treatment

The third area of discussion in this working group is the cleaning and treatment of vacuum chambers, including chemical cleaning, electropolishing, vacuum firing and in-situ bake. There is no universal recipe for cleaning and treatment due to the large variety of material and their intended functions. The available cleaning and treatment is also limited due to the length of the completed chambers. A few points of discussion are listed below. Vacuum firing at high temperature will reduce the hydrogen outgassing but has little effect on ESD. Electro-polishing will re-introduce hydrogen to the bulk therefore should not be done after vacuum firing. The trapped argon or helium during glow discharge cleaning may be desorbed by heavy ion beam scrapping such as the case in RHIC.

Different cleaning recipes were discussed, ranging from DI water rinse to chemical polishing. Due to environmental regulation, most harsh chemicals can't be used for cleaning. The following is what commonly done at a few labs.

Cornell – DI water high pressure rinse, alcohol wipe.
KEKB copper chambers – chemical polishing.
RHIC – high pressure detergent wash then tap water rinse, alcohol wipe.
Tesla (DESY) – DI water rinse until passing the conductance test or optical particle counting.

Different labs use different policy on pre-installation testing depending on the available manpower and schedule, ranging from testing every chamber to testing special beam components only. In-situ bake has also been discussed in the working group. One precaution is in-situ bake can only remove loosely bonded molecules since the equivalent thermal energy at a few hundred degrees is still less than 0.1 eV, small compared with beam induced particle energy. Some thin-foil heaters recently developed for LHC's LSS and experimental beam pipes were found to be very useful for application in a space-limited environment.

Appendix

List of Working Group Participants

- Michel Chanel — CERN
- Ping He — BNL
- Dick Hseuh — BNL
- Roberto Kersevan — ESRF
- Yulin Li — Cornell
- Michael Mapes — BNL
- Mike Seidel — DESY
- Peter Seidl — LBNL
- Ysuke Suetsugu — KEK
- Robert Todd — BNL
- Daniel Weiss — BNL